NSWCDD/TR-92/472

NSWCWODET PARTICIPATION IN SPACE SHUTTLE MISSION STS-46

BY JOHN V. FOLTZ

WEAPONS RESEARCH AND TECHNOLOGY DEPARTMENT

20 JULY 1993

NAVAL SURFACE WARFARE CENTER
DAHLGREN DIVISION • WHITE OAK DETACHMENT
Silver Spring, Maryland 20903-5640

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FOREWORD

The Naval Surface Warfare Center, White Oak Detachment (NSWCWODET), played a role in a recent space shuttle mission, STS-46. NSWCWODET was involved in a materials science experiment conducted in the shuttle cargo bay in which various samples were exposed to the space environment to determine their reactivity with the atmosphere in low earth orbit. The NSWCWODET specimens were materials being evaluated as protective coatings for space and other applications. This report summarizes the experiment and the materials samples.

NSWCWODET participation in this project was sponsored by the Office of Naval Research through the Weapons and Spacecraft Materials Block Program managed by Dr. William Messick.

Dr. Wayne K. Stuckey of the Mechanics and Materials Technology Center of The Aerospace Corporation is acknowledged for enabling NSWCWODET to participate in this experiment. He also provided technical details needed to field the samples and interfaced with Johnson Space Center.

Approved by:

Carl E. Mueller

CARL E. MUELLER, Head
Weapons Materials Division

ABSTRACT

NSWCWODET submitted 27 specimens for space flight STS-46 exposure on the space shuttle. The sample set included various carbide, oxide, and phosphate coatings which are being developed to protect space structural and thermal management components from the atomic oxygen present in low earth orbits, structural carbon-carbon composite material, and a space mirror made of foam metal matrix composite. At this writing, the samples returned from the orbital exposure are being analyzed to determine the degree of reactivity with the space environment.

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INTRODUCTION

In July 1992, the space shuttle program launched STS-46 on an eight-day mission. The flight was intended to accomplish a number of objectives. A retrievable platform carrying various experiments was to be placed in orbit. This platform, called EURECA, would be brought back by another shuttle in 1993. The feasibility of deploying a satellite on a tether was to be explored. A number of cargo bay and middeck payloads, to be activated by the astronauts, were on the manifest.

The Naval Surface Warfare Center, White Oak Detachment (NSWCWODET) participated in the STS-46 mission. One of the cargo bay payloads was an experiment in which samples of various materials would be exposed to the space environment and returned to the ground for postflight analyses of their reaction. NSWCWODET, motivated by a need to develop coatings to protect spacecraft components made of advanced composite materials against the atmosphere of low earth orbit, submitted test samples for this experiment. At this writing, the returned samples are undergoing preliminary analyses. This technical report provides background information about the shuttle flight and documents the materials exposed in the NSWCWODET investigations.

BACKGROUND

The Problem

Materials are affected by the thin upper atmosphere. Many polymers and some metals that returned from the first shuttle flights exhibited significant changes in surface properties attributed to the orbital environment. Susceptibility to atomic oxygen seemed the most plausible explanation for the effects observed.

Early satellite measurements had revealed that the neutral oxygen atom is the predominant species in the upper atmosphere (200 to 600 km), formed at these altitudes through the dissociation of O_2 by ultraviolet radiation. The atom, in the $O(^3P)$ state, is a strong oxidizing agent. Oxidizing effects from laboratory experiments with atomic oxygen were reported in the literature well before the first shuttle flight.[1]

Concentration of oxygen atoms is low at altitudes above 200 km, but a spacecraft suffers a significant incident flux by virtue of its orbital speed. The flux may be estimated from the atomic oxygen density,[2] and the vehicle speed, typically 8 km/sec. Table 1 gives some representative numbers for two altitudes. The fluence, or total number of particles impacting a unit area over an extended exposure, is the time integrated flux. The collision energy of the atom with the spacecraft is 5 eV.

Previous Flight Experiments

In the early 1980s, NASA began developing flight experiments to address the atomic oxygen problem. The approach was to use the shuttle to convey samples to the space environment where they would be exposed in a known manner and then return them to Earth for postflight analyses. The experiments, known as EOIM or Evaluation of Oxygen Interactions with Materials, were characterized by exposures of short duration, typically lasting about 40 hours. In the mid-1980s, NASA also developed the LDEF or Long Duration Exposure Facility to provide information about the effects of much longer exposures. The LDEF was a retrievable platform on which samples were mounted, placed in orbit by one shuttle and recovered months later by another.

In the 1980s, two flights occurred in the EOIM series. EOIM-1 and EOIM-2 evaluations were performed on the fifth (STS-5) and eighth (STS-8) Shuttle missions. The results of the EOIM investigations provide much of the current information regarding reactivities of materials with atomic oxygen in the low earth orbit environment.[2] STS-5 carried about 60 samples and STS-8 over 300. Diagnostic investigations ranging from mass change and surface morphology to surface-chemistry changes were conducted on the exposed surfaces after the flights. Although the exposure times were comparable for both missions, a difference in flight altitudes (300 km for STS-5 compared to 225 km for STS-8) resulted in total accumulated fluences of 1.0×10^{20} and 3.5×10^{20} for STS-5 and STS-8, respectively. Surface recession (or thickness loss) for reactive materials, as determined from change in mass of the specimens, was found to be proportional to the fluence. Qualitatively, the findings are:[2] (1) organics (materials containing only carbon, hydrogen, oxygen, or nitrogen) have high reaction rates; (2) perfluorinated and silicone polymers are more stable than the organics by at least a factor of 50; (3) macroscopically, metals, except for osmium and silver, are stable.

The LDEF was placed into orbit in April 1984. Originally intended to be one year, the exposure actually lasted for almost six years. LDEF contained 57 experiments with over 200 principal investigators and more than 10,000 test samples. While many LDEF studies are still in progress, results to date have given valuable information on long-term performance in orbit. Reference (3) discusses the lessons learned thus far from LDEF.

Dates of these flight experiments are given in Table 2. For additional details about the results consult the Bibliography.

DETAILS OF PRESENT FLIGHT EXPERIMENT

The EOIM-3 experiment was conducted on the 49th overall flight of the space shuttle program as part of the STS-46 mission, summarized in Table 3. Launch and landing were at the Kennedy Space Center in Florida. The director of EOIM-3 is L. J. Leger of the Johnson Space Center. Sample spaces were allocated to the various NASA agencies, the Department of Defense (with the lead laboratory being the Aerospace Corporation), the University of Alabama in Huntsville, The European Space Agency, Canada, and Japan. Overall, more than 1100 samples were submitted. NSWCWODET, the only Navy laboratory to participate, did so under the auspices of the Aerospace Corporation.

NSWCWODET investigators need to know the reaction rates of certain materials which are candidates for protective coating systems. Samples consisted of flat substrates on which experimental coatings were deposited. The list of coatings and substrates is given in Table 4. Two materials were tested as monolithic discs.

The substrates were of AXF-5Q polycrystalline graphite made by Poco Graphite, Inc., carbon-carbon composite material (C-C) made by Kaiser Aerotech, or silicon carbide aluminum metal matrix composite material made by Advanced Composite Materials Corporation. Polycrystalline graphite was chosen as a substrate for basic research materials because it is inexpensive, readily polished, and has a low thermal expansion coefficient similar to the coatings placed on it. Carbon-carbon composite is being developed for spacecraft applications and is representative of an engineering structural material. Carbon-based materials in general will require an undercoat for atomic oxygen protection and an overcoat for thermal control. The metal matrix composite substrate is actually a prototype space mirror, consisting of a foam SiC/Al core with a face sheet of optical grade SiC/Al.

NSWCWODET samples were fabricated in the form of small discs 1/2 inch in diameter. The fixture which holds samples during the space exposure is drawn in Figure 1. Control samples were fabricated for comparison with exposed materials. When the sample size permited, two samples of each material were flown in the same fixture: the evaluation sample flown face up and exposed to the oxygen stream and the flight control sample, mounted face down in the tray. The thickness of the SiC/Al mirror prohibited a flight control sample for this material.

All samples were photographed and weighed before assembly into the flight trays. Figure 2 shows a sample being loaded. Fully loaded ambient temperature tray no. 12 holding the 82 samples from NSWCWODET and Aerospace Corporation is shown in Figure 3. Figure 4 is a template to identify the samples (NSWC WODET samples are no. 41 and no.'s 45-64). The assembled tray and all associated hardware were placed in a vacuum chamber on a table maintained at 65°C and outgassed for 72 hours.

Three trays were designed with heaters to hold samples in orbit isothermally at 60°C, 120°C, or 200°C. The purpose is to obtain an assessment of the effect of temperature on material reaction rates. TiC and VC coating samples were exposed on each of the three elevated temperature trays. Flight control samples were not used.

The EOIM-3 qualification hardware is shown mounted on a table for a vibration test, Figure 5. Fifteen ambient temperature trays (sans specimens) are in the foreground of the picture. The mass spectrometer is indicated by the arrow. The three elevated temperature trays are visible at the top of the assembly on the right hand side. The flight hardware is a duplicate of this assembly and is mounted on a truss support structure located in the aft region of the orbiter cargo bay, Figure 6. This arrangement is intended to minimize reflection of the incoming atomic oxygen flux and provide samples with an unobscured view for direct impingement.

In previous flight experiments calculations based on models of the earth's atmosphere were used to estimate atomic oxygen flux during the exposure period, leading to an uncertainty in fluence on the order of 15 to 20 percent. Reaction rate determinations from EOIM-3 are expected to be more accurate than those yielded by earlier flight evaluations due to the use of an ion-neutral mass spectrometer to measure the incoming atomic oxygen atom flux *in-situ*.

RESULTS

A high percentage of the STS-46 secondary payloads successfully accomplished their purpose. The EURECA retrievable platform was placed in orbit. Attempts to deploy the Tethered Satellite System were not fully successful due to a problem with the tether reel mechanism. The crew was able to cast the satellite out to only a small fraction of the intended length. The EOIM-3 experiment was executed largely as intended, although the start was delayed. The experiment was initiated by Atlantis' crew on the sixth day of the flight. The shuttle was lowered to an altitude of 230 km and oriented nose-to-earth with the cargo bay facing into the velocity vector. The cargo bay doors were opened and the samples were exposed for 42 hours. The exposure provided a calculated atomic oxygen fluence of 1.99×10^{20} atoms/cm^2, based on values of solar activity predicted before the flight. Results of the mass spectrometer measurements were not available at this writing.

After return to earth, the samples were unloaded and the trays distributed to the principal investigators. This process took approximately three weeks. Preliminary postflight analyses of all DOD samples were conducted at the Aerospace Corporation. Initially, all samples were photographed, dessicated, and weighed. Scanning electron microscopy observations were recorded.

Photographs of four samples taken postflight are shown in Figure 7. The experimental sample and the flight control sample are presented together for comparison. The test results confirm that the baseline carbon carbon composite was, as expected, susceptible to atomic

oxygen attack. Etching of the uncoated composite is clearly visible in Figure 7(a) as a darkened ring inside the area protected by the flange which held the sample in place. Overall, eight of the other twenty test samples on the ambient tray exhibited obvious effects of exposure to the space environment in a visual examination. Pictures showing the results for three of the test coatings are in Figure 7(b), (c), and (d).

FUTURE WORK

The samples will be returned to NSWCWODET in early 1993 for in-house detailed analyses which will be reported by the principal investigators. Microscopy (electron or optical) will be the initial diagnostic tool. Other analytical methods such as x-ray photoelectron spectroscopy, Rutherford backscattering spectrometry, or atomic force microscopy will be employed as appropriate. Integrated scattering and diffuse reflectance measurements are techniques useful in characterizing the space mirror, which was intitially polished to a specular finish.

SUMMARY

NSWCWODET submitted 27 samples for a materials science experiment aboard the space shuttle on mission STS-46. The NSWCWODET samples are being evaluated as protective coating materials by determining their reactivity with the low earth orbit atmosphere in a space flight exposure. At the writing of this report, the space flight exposure has been successfully accomplished and the samples returned to earth for analyses. Preliminary results indicate some of the materials reacted significantly with the space environment.

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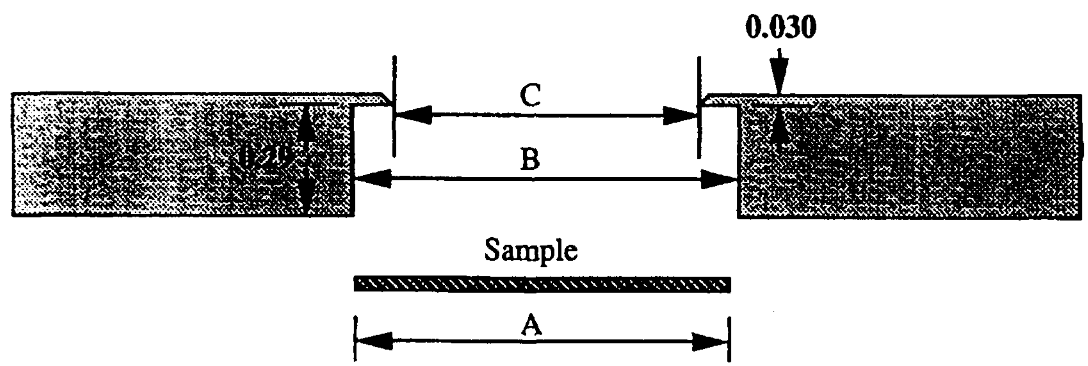


	A	B	C
1" Sample	$0.995 \begin{smallmatrix}+0.000\\-0.005\end{smallmatrix}$	1.020 ± 0.005	0.813 ± 0.010
1/2" Sample	$0.495 \begin{smallmatrix}+0.000\\-0.005\end{smallmatrix}$	0.520 ± 0.005	$0.375 \pm .010$

FIGURE 1. DIMENSIONS OF SAMPLE HOLDER ON CARRIER TRAY

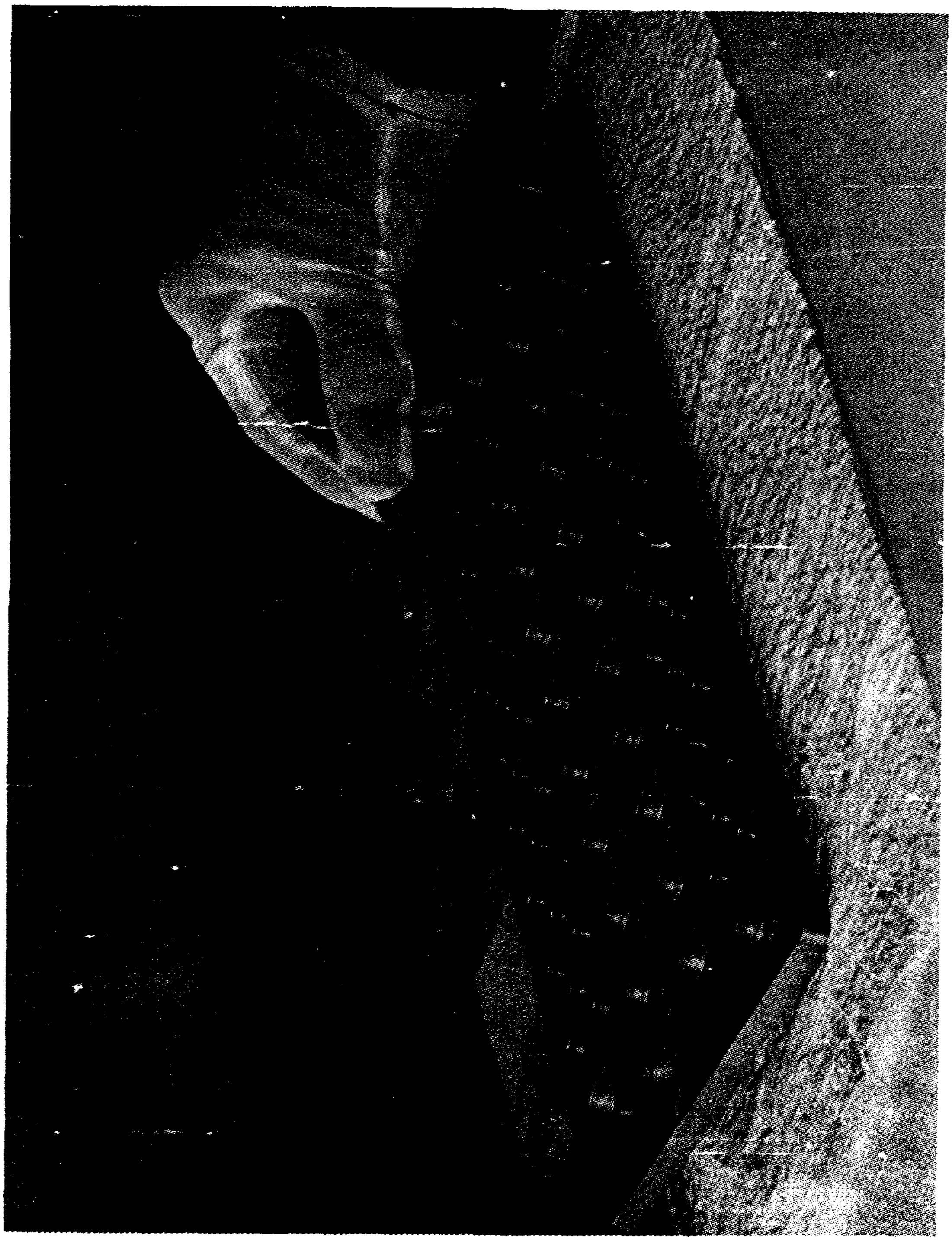


FIGURE 2. SAMPLE CARRIER TRAY NO. 12 (UNLOADED)

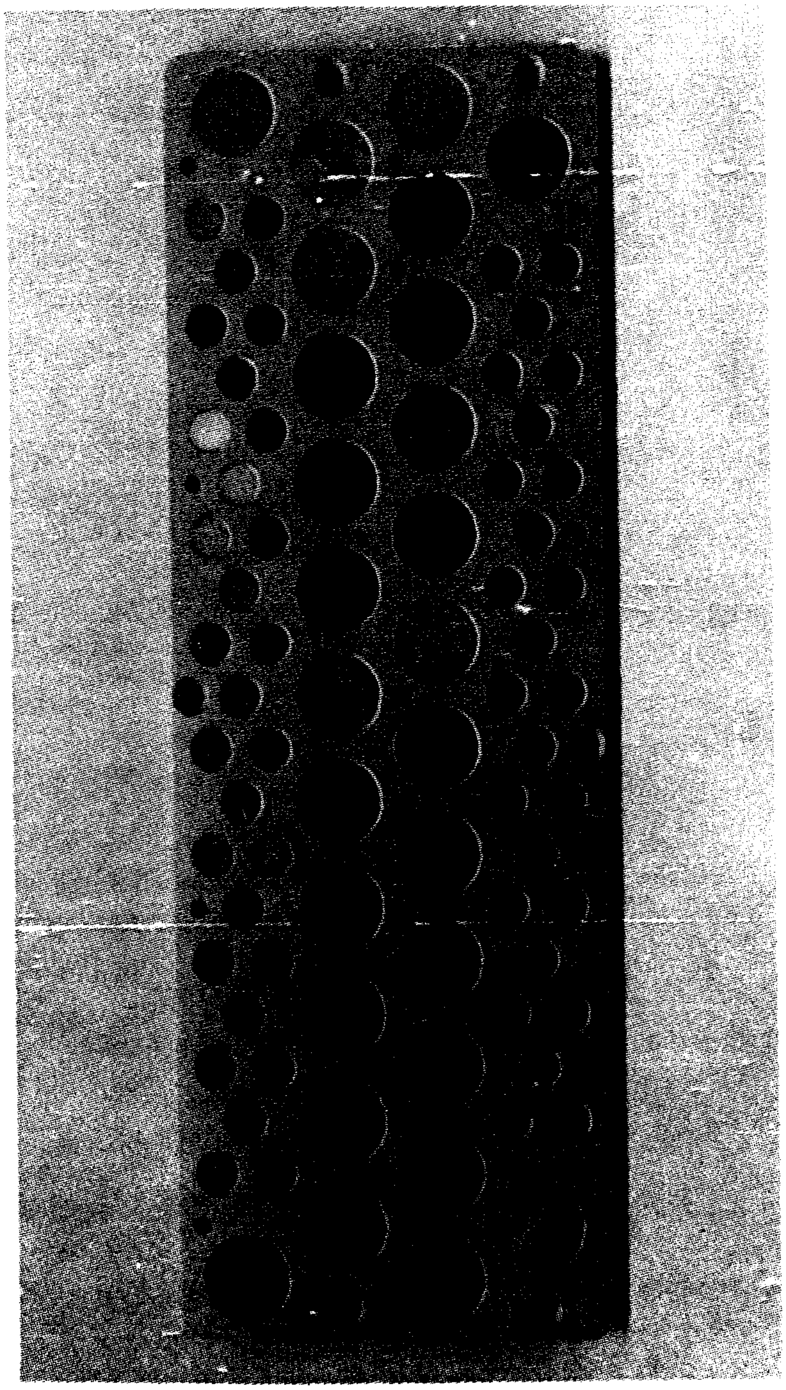
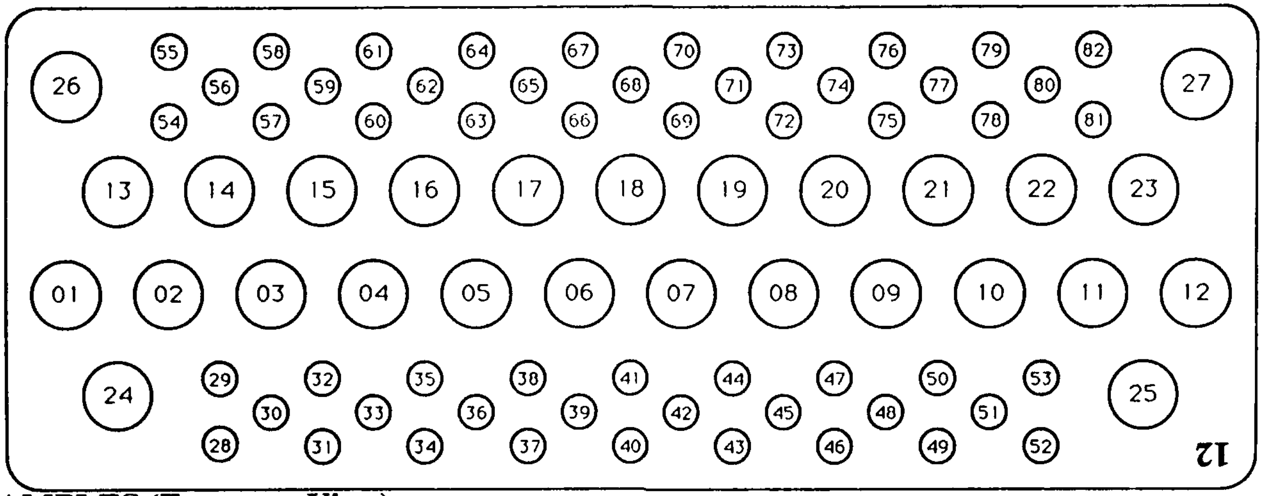


FIGURE 3. SAMPLE CARRIER TRAY NO. 12 (LOADED)



SAMPLES (Exposure View):

01 Al2O3/BN/SiO2	18 Carbon Film/Ag	35 TiO2/Fused Silica	52 V/POCO	69 6FDA+APB-Spray
02 Al2O3/BN/SiO2	19 Black Kapton-old	36 TiO2/C-C	53 Cr/POCO	70 BTDA+4,4ODA
03 BN/SiO2	20 Black Kapton-new	37 TiO2/C-C	54 Si&SiC/C-C	71 Liquid Crystal Epoxy
04 BN/SiO2	21 Ge/Kapton	38 TiO2/POCO	55 SiO2/Si-SiC/C-C	72 Epon 825/PDA
05 BN/Al/SiO2	22 Ag Interconnect	39 TiO2-SiO2/Si	56 Al2O3/Si-SiC/C-C	73 Vectra 4950 Polyester
06 BN/Al/SiO2	23 Ag Interconnect	40 Ni/SiC Al	57 Rh/Si-SiC/C-C	74 XYDAR SRT 300
07 SiO2/TiN/SiO2	24 Coated SiC-Al	41 SiC Mirror	58 ZrP2O7/C-C	75 Sperex Black
08 SiO2/TiN/SiO2	25 LDEF A276TE	42 P75/934	59 SiO2/ZrP2O7/C-C	76 Sperex SP101
09 LDEF Al TE	26 6FDA+APBspin	43 AS4/PEEK	60 SiP2O7/C-C	77 Rh/Mo
10 LDEF Z306	27 6FDA+APBspray	44 6FDA+DDSO2	61 SiO2/SiP2O7/C-C	78 954-3 Cyanate Ester
11 LDEF S13GLOLE	28 Fluorinated 7940	45 TiC/POCO	62 Carbon-Carbon	79 934 Epoxy
12 LDEF S13GLOTE	29 Fluorinated 7940	46 VC/POCO	63 AlPO4/C-C	80 High Temp. Adh.
13 MoS2 on Steel	30 OCLI 3	47 TiB2/POCO	64 SiO2/AlPO4/C-C	81 CV1144 on Gr/Ep
14 MoS2 on Steel	31 OCLI 4	48 TiC/POCO	65 Carbon Film (Si)	82 AO Resistant Polyimide
15 MoS2 on Steel	32 ZnS/Si	49 NiAl	66 Carbon Film (Si)	
16 MoS2 on Steel	33 ZnS/Si	50 NiBe/POCO	67 BFDA+4BDAF	
17 Carbon Film/Ag	34 TiO2/Si	51 Ti2Be17/POCO	68 6FDA+APB-Spin	

FIGURE 4. SAMPLE IDENTIFICATION TEMPLATE FOR TRAY NO. 12

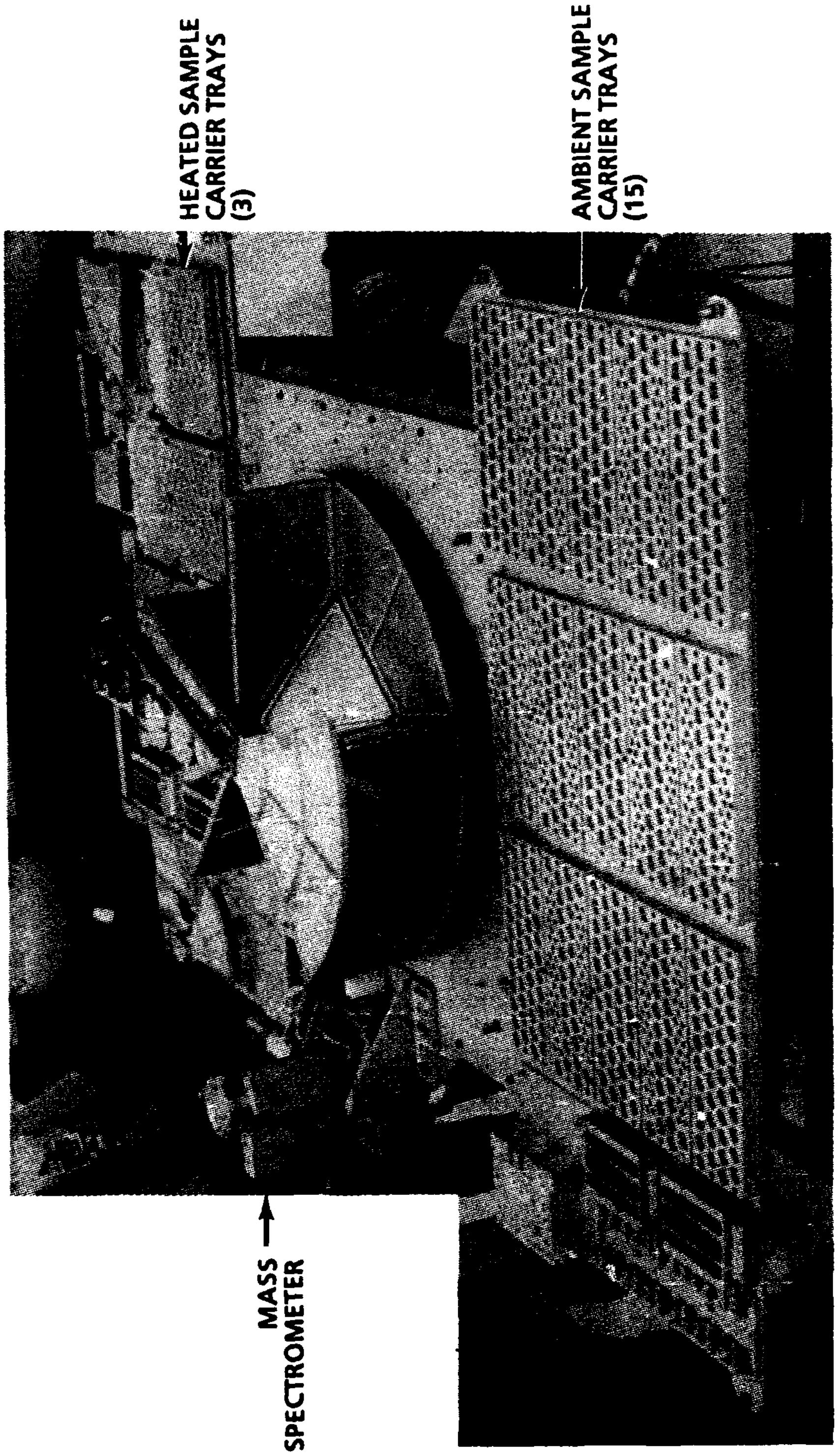


FIGURE 5. QUALIFICATION TEST SETUP FOR EOIM-3

National Aeronautics and Space Administration S92 42288 Lyndon B. Johnson Space Center Houston, Texas 77058

FIGURE 6. LOCATION OF EOIM-3 ON SHUTTLE

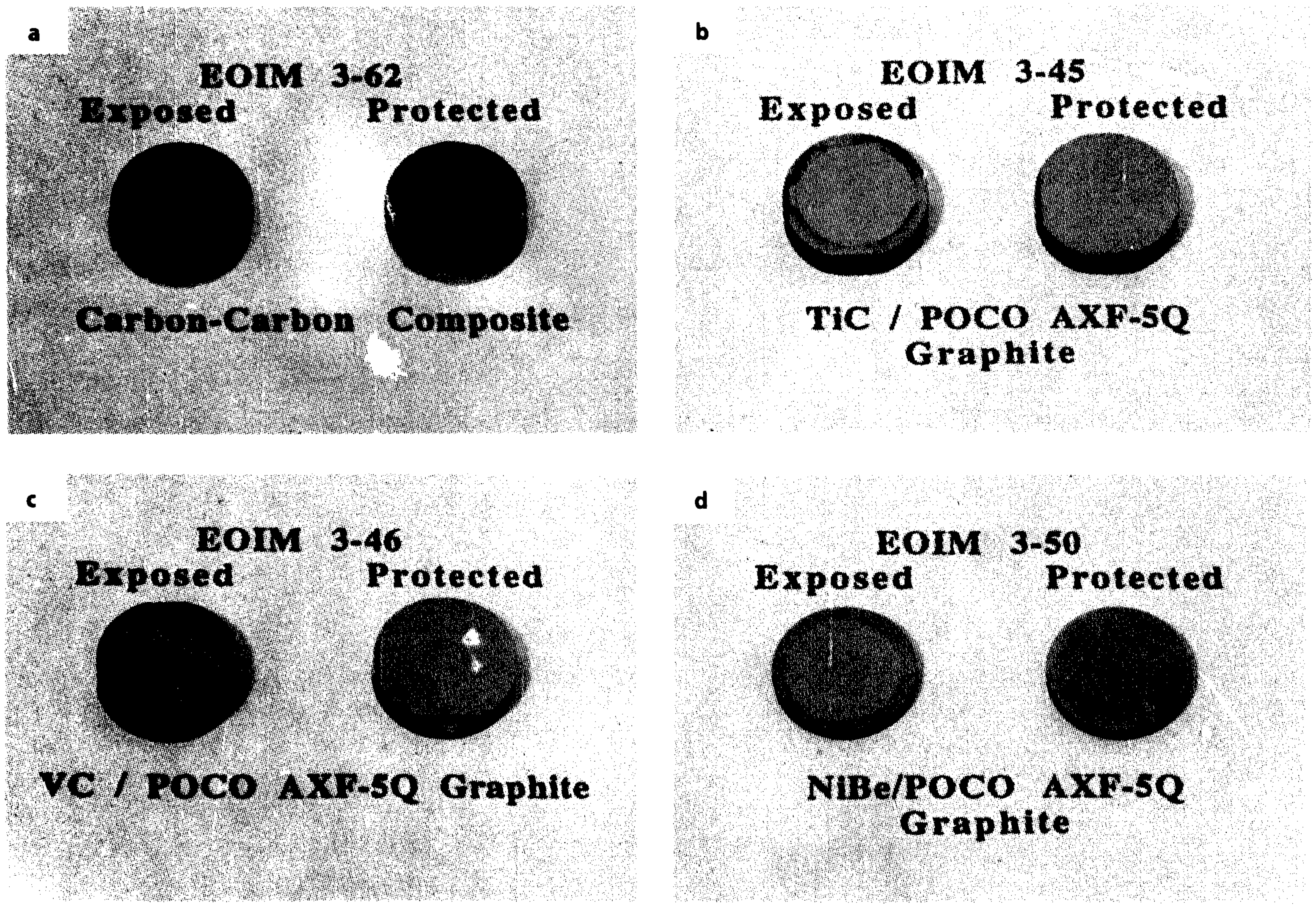


FIGURE 7. FOUR SAMPLES EXPOSED TO THE SPACE ENVIRONMENT

TABLE 1. ATOMIC OXYGEN FLUX VERSUS ALTITUDE*

ALTITUDE (km)	DENSITY (atoms cm^{-3})	FLUX (atoms cm^{-2} sec^{-1})
200	5×10^9	4×10^{15}
600	5×10^6	4×10^{12}

* Atomic oxygen density from Reference 2.

TABLE 2. DATES OF EOIM-1, EIOM-2, AND LDEF

EXPERIMENT	FLIGHT DATE
EOIM-1	11 Nov 1982 - 16 Nov 1982
EOIM-2	30 Aug 1983 - 5 Sep 1983
LDEF	7 Apr 1984 (Launch) 12 Jan 1990 (Recovery)

TABLE 3. STS-46 MISSION SUMMARY

FLIGHT DATE:	31 Jul 1992 - 8 Aug 1992
ORBITER:	ATLANTIS (Twelfth Flight)
MISSION DURATION:	8 Days
MILES TRAVELED:	3.5 Million
ORBITS OF EARTH:	126
ORBITS:	230 NM X 230 NM (EURECA*) 160 NM X 160 NM (TSS**) 124 NM X 124 NM (EOIM-3)
CREW:	Loren J. Shriver (Commander) Andrew M. Allen (Pilot) Claude Nicollier Marsha S. Irvins Jeffrey A. Hoffman Franklin R. Chang-Diaz Franco Malerba

* European Retrievable Carrier Platform
** Tethered Satellite System

TABLE 4. NSWC SAMPLES ON AMBIENT TEMPERATURE TRAY NUMBER 12

COATING	UNDERCOAT	SUBSTRATE	NSWC POINT OF CONTACT
TiC	--	POCO	M. Opeka
VC	--	POCO	M. Opeka
TIB2	--	POCO	M. Opeka
TiC	--	POCO	M. Opeka
--	--	NiAl	M. Opeka
NiBe	--	POCO	M. Opeka
Ti2Be17	--	POCO	M. Opeka
V	--	POCO	M. Opeka
Cr	--	POCO	M. Opeka
Si-SiC*	--	C-C	M. Opeka
Rh	Si-SiC*	C-C	M. Opeka
Si02	Si-SiC*	C-C	M. Opeka
Al203	Si-SiC*	C-C	M. Opeka
--	--	C-C	M. Opeka
ZrP207	--	C-C	J. Zaykoski
Si02	ZrP207	C-C	J. Zaykoski
SiP207	--	C-C	J. Zaykoski
Si02	SiP207	C-C	J. Zaykoski
AlP04	--	C-C	J. Zaykoski
Si02	AlP04	C-C	J. Zaykoski
Ni	--	SiC/Al	J. Foltz

* Si-SiC is a co-deposited material.

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13. ABSTRACT *(Maximum 200 words)*

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